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CHLORIDE ION CONDUCTIVITY IN A PLASTICIZED QUATERNARY
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L C HARDY ET AL. 22 NOV 83 TR008 N00014-80-C-0532

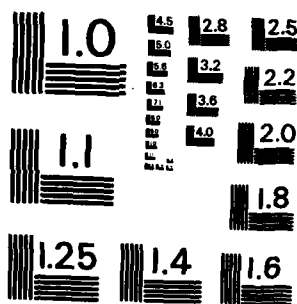
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Chloride Ion Conductivity in a Plasticized Quaternary

Ammonium Polymer

by

L. C. Hardy and D. F. Shriver

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Chloride Ion Conductivity in a Plasticized Quaternary Ammonium Polymer

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Solvent-free polymer electrolytes^{1,2} have generated much interest owing to their possible use in high energy density batteries. Most research in this area has focused on alkali metal ion conduction in solid electrolytes formed by alkali metal salts and poly(ethylene oxide)³ or poly(propylene oxide).² Although cation transport has been substantiated by use of reversible cation electrodes, i.e. alkali metals,⁴⁻⁶ intercalates,¹ and sodium amalgam,⁷ charge transport by anions may be substantial.^{5,6} Here we report the first studies of fast anion conduction in solid polymer electrolytes, poly(diallyldimethylammonium chloride), DDAC, plasticized with poly(ethylene glycol), PEG (average MW = 300). In these anhydrous electrolytes, the positive charge is anchored in the polymer backbone. Thus the DC electrical properties displayed by these conductors are clearly attributable to the anion.

An important motivating factor for the use of DDAC is that the positive quaternary nitrogen is surrounded by four alkyl groups, thus separating the opposite charges and reducing tight ion pairing. Tight ion pairing has been shown to significantly reduce the mobility of charge carriers in solid polymer electrolytes.⁸

Use of plasticizers in polymers is known to increase polymer chain flexibility, increase free volume, and decrease the glass transition temperature, T_g . These changes are also known to increase ionic conductivity in solid electrolytes.^{9,10} Therefore, the present research was designed to investigate the possibility that plasticizers might increase the ionic conductivity of the polyelectrolyte DDAC.

The conductivity, σ , of pure, brittle DDAC at 25°C was not measurable on our instruments, which indicates a conductivity much less than $10^{-9} \Omega^{-1}\text{cm}^{-1}$. A number of esters and ethers were tested for compatibility as plasticizers. Brittle, cloudy-white films resulted when mixtures of DDAC with diisodecylphthalate, dibutylphthalate, dibutylsebacate, tetraethyleneglycoldimethylether, poly(ethylene oxide), or poly(vinyl alcohol) (88% hydrolyzed) were cast from methanol or methanol/water solutions at 25 or 60°C. Segregation of the plasticizer also occurred in some cases. The only plasticizer found to be effective was PEG (av. MW = 300). Since the similar compound tetraethylene glycol dimethylether was incompatible, the hydroxyl groups on the polyether must play an important role in the plasticizing interaction. This hypothesis is substantiated by IR spectroscopy which shows that the O-H stretch for pure PEG is lowered from $\sim 3400 \text{ cm}^{-1}$ to $\sim 3240 \text{ cm}^{-1}$ when mixed with DDAC in a ratio of one hydroxyl group to two chloride ions. This behavior suggests hydrogen-bonding to the more electronegative chloride ion in preference to hydrogen-bonding with the oxygen such as found in pure PEG. Evidence for similar plasticizing behavior has been reported for sodium styrene sulfonate groups and glycerol.¹¹

Standard inert atmosphere techniques were used in the preparation and study of these electrolytes because water appears to displace the glycol from.

the plasticized polymer. After preparation from nominally dry materials, the last traces of water were removed by vacuum drying of films. This treatment leads to the disappearance of the H-O-H deformation at $\sim 1630\text{ cm}^{-1}$ in the infrared spectra. This band reappears when the sample is exposed to atmospheric moisture. Films with 0.32, 0.48 and 0.65 weight fractions of PEG were made and these correspond to mole ratios of chloride ion to hydroxyl group of 2, 1 and 0.67, respectively. These films are clear and flexible at room temperature. Differential scanning calorimetry ($20^\circ\text{C}/\text{min}$) indicates that T_g is $-37 \pm 2^\circ\text{C}$ for all three mole ratios. No endotherms are observed for the melting of pure PEG in these electrolytes. Polarized optical microscopy also indicates that the films are amorphous.

AC complex impedance data collected over the frequency range of 10 Hz-500 kHz with platinum ion-blocking electrodes were used to calculate bulk conductivity of samples. The variable temperature electrical conductivity data yield curved plots of $\ln(\sigma T)$ vs. $1/T$, Figure 1. This behavior, which is similar to that of diffusion of small molecules through polymers, has been seen previously for other polymer electrolytes.^{2,12} Transport of ions in these materials may be described by WLF-type equations rather than the Arrhenius law.^{2,10,13} Conductivity increases as the amount of plasticizer in the electrolyte increases, see Table 1.

Plots of ϵ' and ϵ'' vs. frequency with ion-blocking electrodes are shown in Figure 2. As frequency decreases, interfacial polarization of the ionic conductor increases which results in a continuously increasing dielectric constant as has been predicted by theory,¹⁴ and has been reported for inorganic solid electrolytes.^{15,16} Therefore, the reported dielectric constants

are due to double layer capacitances rather than molecular dipoles. According to the theory,¹⁴ at low frequencies the internal field of the electrolyte equally opposes the applied field and no further polarization occurs so the ϵ' curve flattens out. The beginning of this behavior is seen in the high temperature plot. The absence of maxima in the ϵ'' plots, above 10 Hz, in conjunction with other data indicates that the frequency response over the frequency range studied is due to ion motion rather than molecular relaxations such as the rotation of an -OH group. High dielectric constants also have been reported for solid electrolytes formed between poly(ethylene oxide) and alkali metal salts.¹⁷

Complex impedance plots are typical of those seen for ionic conductors with ion-blocking electrodes, Figure 3a. In particular, the spur found at low frequencies is ascribed to double layer capacitance in a cell with an ion blocking electrode configuration.¹⁸

To provide a definitive assignment of the low frequency spur, calomel electrodes were employed in a second set of complex impedance experiments. The spur seen previously for ion-blocking electrodes is now converted to an arc, Figure 3b. According to impedance/frequency response theory,¹⁸ this arc can be ascribed to charge transfer resistance at the chloride ion reversible electrode. This type of response is well characterized for other polymer electrolyte systems with cation reversible electrodes.^{5,6}

The present work demonstrates that conductivity due to anion transport in a solvent-free polymer system can be on the same order of magnitude as that found for poly(ethylene oxide)-alkali metal salt systems. The electrical properties characterized in the present system may be attributed solely to the anion since the positive counter charge is anchored in the polymer backbone.

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Table 1

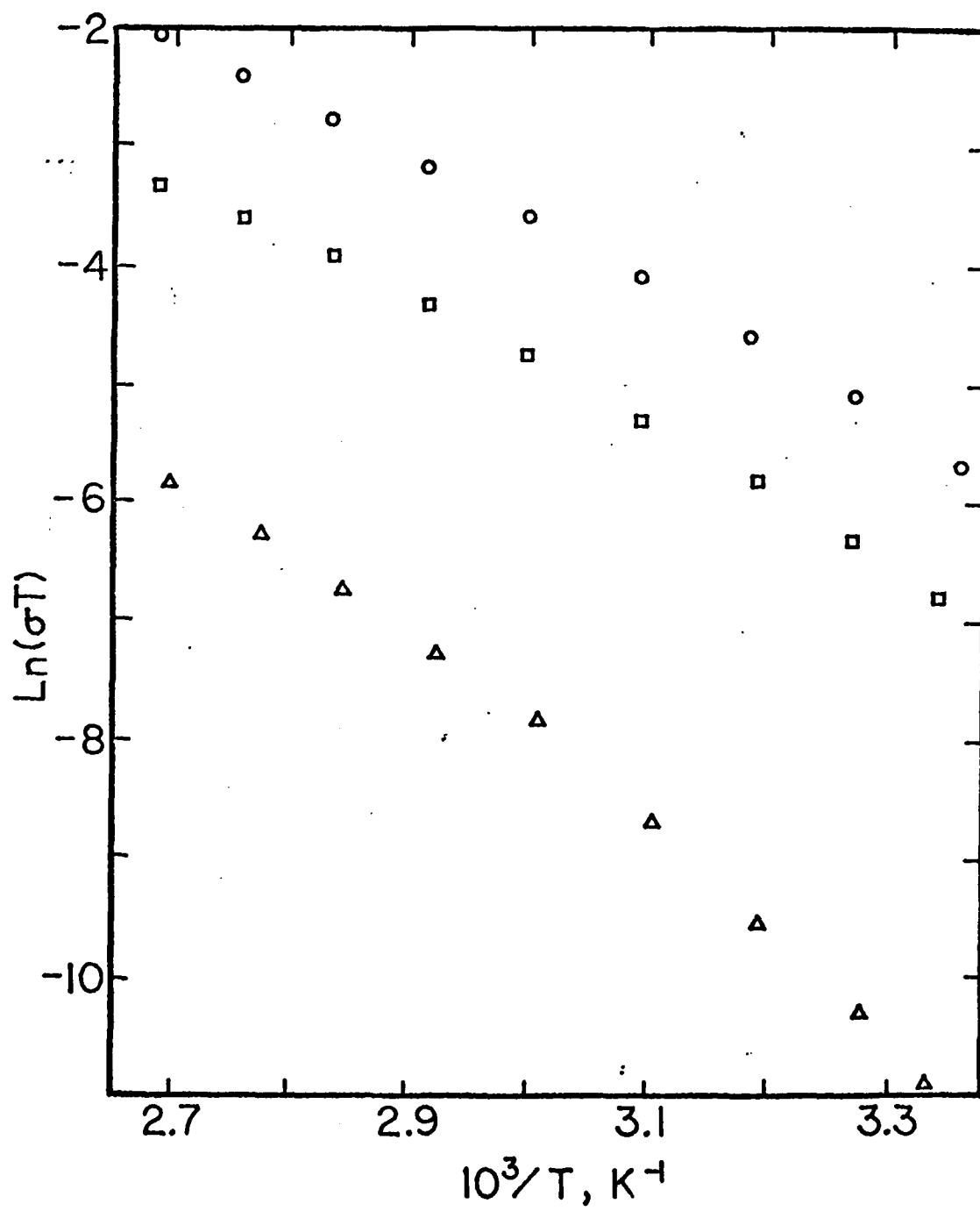
| Mole ratio Cl ⁻ :hydroxyl | Conductivity ($\Omega^{-1} \text{ cm}^{-1}$) | |
|---|--|--------------------|
| | 26°C | 98°C |
| 2:1 | 7×10^{-8} | 1×10^{-5} |
| 1:1 | 4×10^{-6} | 1×10^{-4} |
| 0.67:1 | 1×10^{-5} | 3×10^{-4} |

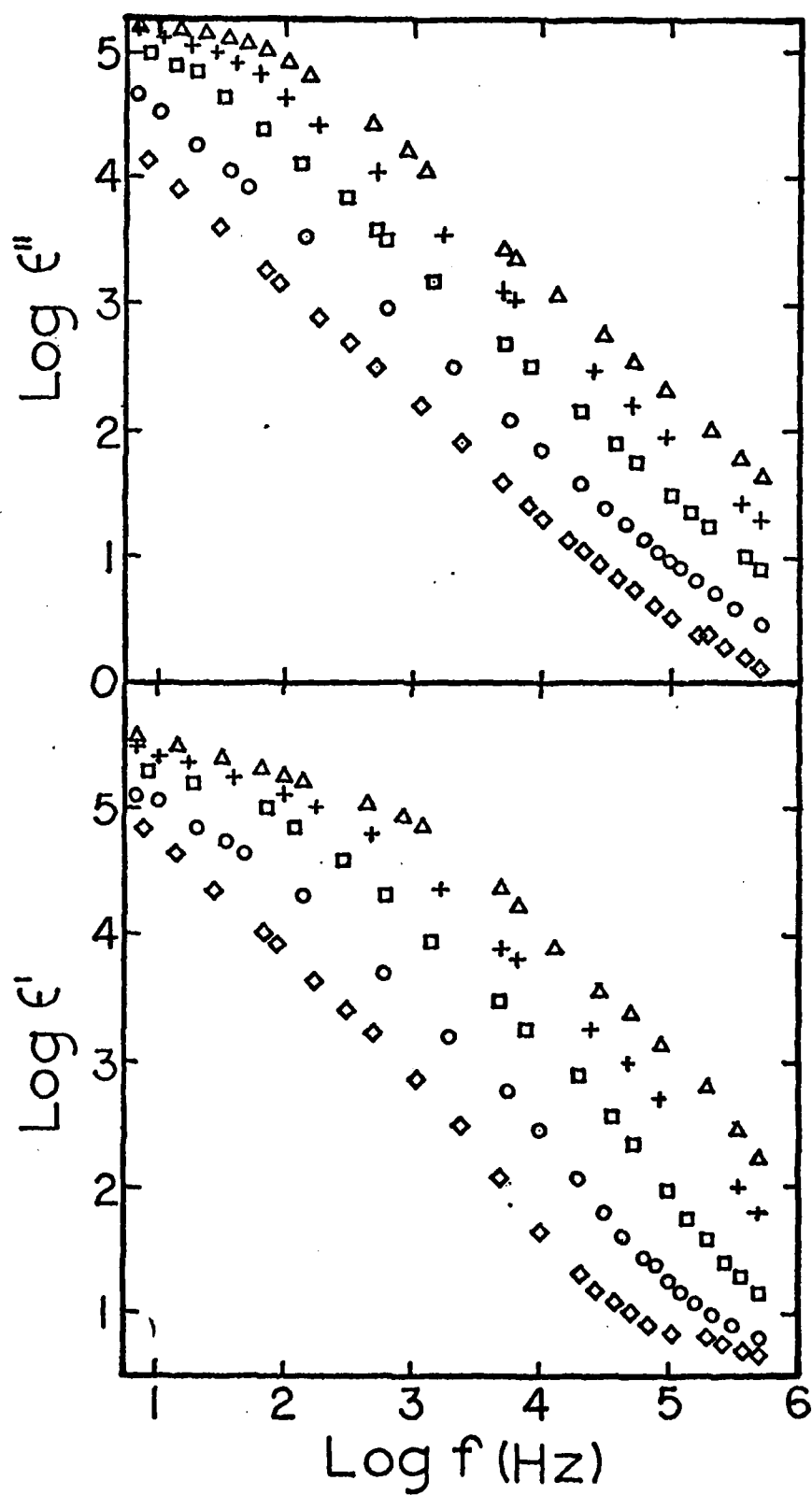
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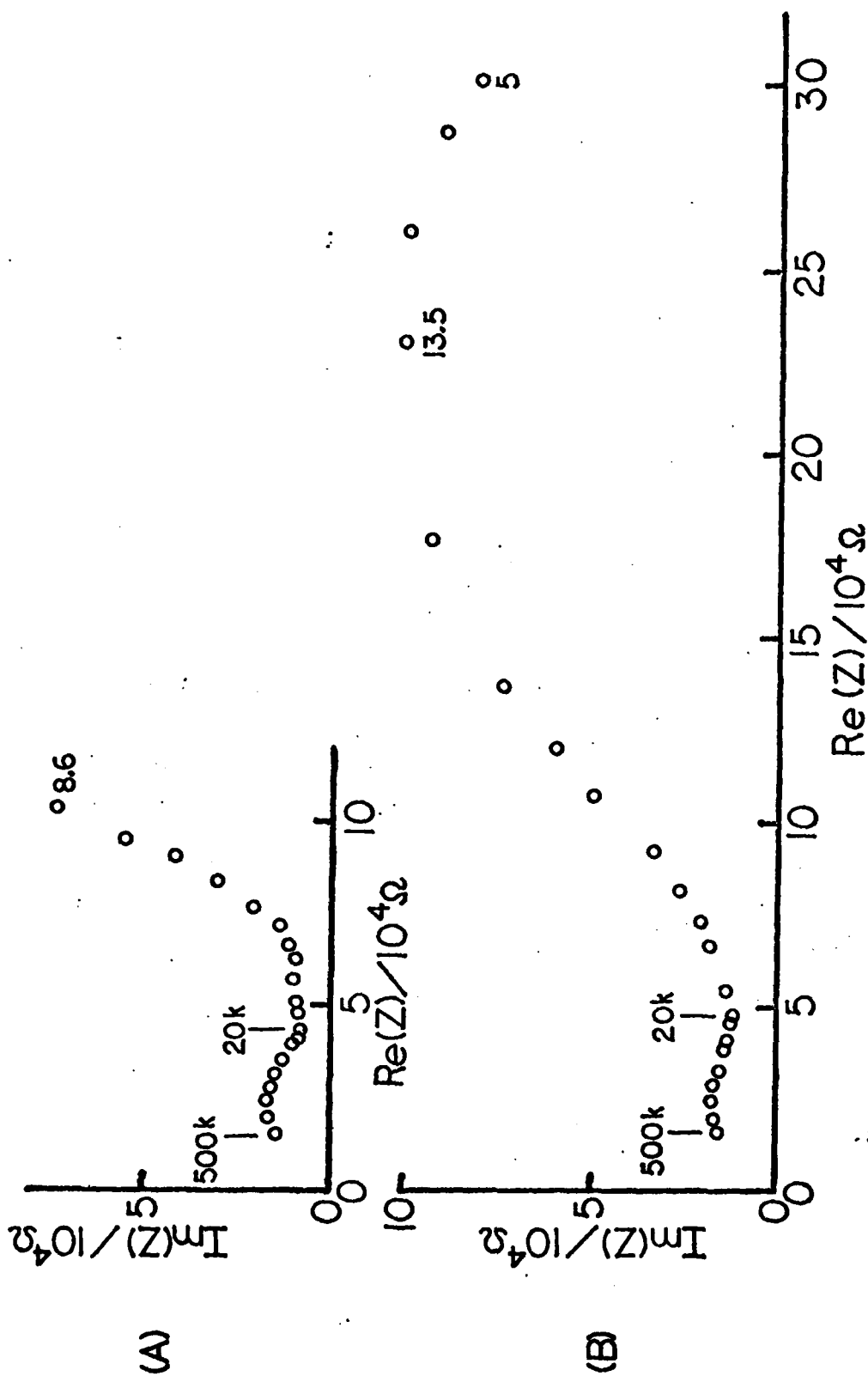
Figure 1. Ionic conductivity, $\sigma(\Omega^{-1} \text{ cm}^{-1})$, versus temperature plotted as $\ln(\sigma T)$ vs. $1000/T$, for various mole ratios of chloride ion in polymer to hydroxyl group in plasticizer: Δ , 2:1; \square , 1:1; \circ , 0.67:1.

Figure 2. Plots of log dielectric constant, ϵ' , and log dielectric loss, ϵ'' , vs. log frequency in Hertz for the 2:1 chloride to hydroxyl electrolyte at various temperatures: \diamond , 26°C; \circ , 41°C; \square , 60°C; $+$, 79°C; Δ , 98°C.

Figure 3. Plots of the imaginary part vs. the real part of the impedance (Z) for the 2:1 chloride to hydroxyl electrolyte at 60°C. Frequencies (5 Hz - 500 kHz) are listed next to the data points. a) With Pt electrodes. b) With $\text{Hg}/\text{Hg}_2\text{Cl}_2$, calomel, electrodes.







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